

METRIC

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MILITARY SPECIFICATION
STABILIZER ADDITIVE, DIESEL FUEL

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This military specification covers a stabilizer additive for use in diesel fuels meeting the requirements of WV-F-800 which are intended for intermediate (6-24 months) or long-term (25-60 months) storage. Typical applications for this additive are to pre-positioned fuel and equipment, vehicles subject to storage or infrequent use, and to bulk fuel procured for both intermediate and long-term storage (see 6.1).

1.2 Classification. Stabilizer additives shall be of the following types as described in 1.2.1 and 1.2.2:

- Type I - One-package stabilizer additive.
- Type II - Two-package stabilizer additive.

The performance requirements for types I and II are identical.

1.2.1 Type I description. One-package stabilizer additives are those in which all the required components are present in a single homogeneous solution. Only one container is required for complete fuel treatment.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: USA Belvoir Research, Development, and Engineering Center, ATTN: STREE-TSE, Fort Belvoir, VA 22060-5606 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

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1.2.2 Type II description. Two-package stabilizer additives are those in which the required components are divided between two containers, each containing one or more components in a homogeneous solution. Both containers are required for complete fuel treatment.

1.2.3 Part numbering. Stabilizer additives of the following types, components, and sizes shall be numbered as shown below. A complete Type II stabilizer additive shall contain all five components within its two parts, as shown in the last two examples of part numbers.

M53021	-1	-	05	Example of type I part number: M53021-1-05
M53021	-2	-B	55	Example of type II part number: M53021-2-B55
M53021	-2	-ACDM	55	Example of type II part number: M53021-2-ACDM55

Container size, US gallons.

Components (type II only).

- A - Antioxidant
- B - Biocide
- C - Corrosion inhibitor
- D - Dispersant
- M - Metal deactivator

- 1 - Type I, one-package stabilizer additive.
- 2 - Type II, two-package stabilizer additive.

Specification number.

Prefix.

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications and standards. The following specifications and standards form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of these documents shall be those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation.

SPECIFICATIONS

FEDERAL

- | | |
|----------|--------------------------------------|
| TT-S-735 | - Standard Test Fluids, Hydrocarbon. |
| VV-F-800 | - Fuel Oil, Diesel. |

STANDARDS

FEDERAL

- FED-STD-313 - Material Safety Data Sheets, Preparation and Submission of.
- FED-STD-791 - Lubricants, Liquid Fuels and Related Products; Methods of Testing.

MILITARY

- MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.
- MIL-STD-290 - Packaging of Petroleum and Related Products.

2.1.2 Other Government publication. The following other Government publications forms a part of this specification to the extent specified herein. Unless otherwise specified, the issue shall be those in effect on the date of the solicitation.

DEPARTMENT OF LABOR (DOL)

Occupational Safety and Health Administration (OSHA)

- 29 CFR 1910.1200 - Hazard Communication.

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, DC 20402.)

(Copies of specifications, standards, and publications required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.2 Other publications. The following document(s) form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted shall be those listed in the issue of the DODISS specified in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS shall be the issue of the non-Government documents which are current on the date of the solicitation.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS (NACE)

- NACE TM-01-72 - Antirust Properties of Petroleum Product Pipeline Cargoes.

(The test method listed above may be ordered from the National Association of Corrosion Engineers, P.O. Box 218340, Houston, TX 77218.)

SOCIETY OF INDUSTRIAL MICROBIOLOGISTS (SIM)

SIM Special Publication No. 2 - Proposed Procedure for the Screening of Microbial Inhibitors in Hydrocarbon/Water Systems.

(This publication is currently out of print. Reproduced copies are available from the USA Belvoir Research, Development, and Engineering Center, ATTN: SIRBE-VF, Ft. Belvoir, VA 22060-5606.)

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

- D 93 - Flash Point by Pensky-Martens Closed Tester.
- D 445 - Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity).
- D 482 - Ash from Petroleum Products.
- D 664 - Neutralization Number by Potentiometric Titration.
- D 974 - Neutralization Number by Color-Indicator Titration.
- D 1298 - Relative Density (Specific Gravity) or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method.
- D 2274 - Oxidation Stability of Distillate Fuel Oil (Accelerated Method).
- D 4057 - Manual Sampling of Petroleum and Petroleum Products.
- D 4625 - Distillate Fuel Storage Stability at 43 °C (110 °F).
- STP 509A - Single Cylinder Engine Tests for Evaluating the Performance of Crankcase Lubricants.

(The test methods listed above are included in Volumes 05.01, 05.02, and 05.03 of the Annual Book of ASTM Standards and are available individually. Special Technical Publications (STP) are available individually. Applications for copies of all ASTM publications should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

(Non-Government standards and other publications are normally available from the organizations which prepare or which distribute the documents. These documents also may be available in or through libraries or other informational services.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, (except for associated detail specifications, specification sheets or MS standards), the text of this specification shall take precedence. Nothing in this specification, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Qualification. The stabilizer additives furnished under this specification are for use in diesel fuels. The stabilizer additives shall be a product that has passed the applicable qualification requirements of 3.1.1 or has been listed on or approved for listing on the applicable qualified products list. Tentative approval for listing on the qualified products list shall be granted

pending successful completion of all requirements of 3.1.1 except for an ongoing 12 month storage stability test (3.13). Full approval shall be granted upon successful completion of storage stability test. Failure to pass the storage stability test shall be cause for withdrawal of approval.

3.1.1 Qualification requirements. All approved stabilizer additives shall meet the requirements of 3.2 through 3.22 to be qualified for use in diesel fuels.

3.2 Identification qualification data. The following properties of the finished stabilizer additive shall be determined but not limited during qualification: density at 15 °C, viscosity at 0 °C and 40 °C, neutralization number, pHr, and type of metallic constituent, if present (see 4.6.1). The permissible production variation of individual properties will be established at the time of qualification by mutual agreement between the manufacturer and the qualifying activity. Individual batches of stabilizer additive subsequently subjected to quality conformance inspections shall conform to the established range of properties. The ranges shall not adversely affect any of the stabilizer additive performance characteristics.

3.3 Materials. The stabilizer additive supplied under this specification shall consist of petroleum-soluble compounds which perform the following functions:

- a. Antioxidant.
- b. Biocide.
- c. Corrosion Inhibitor.
- d. Dispersant.
- e. Metal deactivator.

The composition of the finished stabilizer additive is not limited, except as specified in 3.3.2 and 3.3.3, but is subject to review by the qualifying activity in order to assure service compatibility with previously qualified products.

3.3.1 Recommended effective concentration. Each contractor of stabilizer additive shall designate a recommended effective concentration at which the product should be used in diesel fuel. The recommended effective concentration shall be expressed in both grams per cubic meter (g/m^3) and in quantity per 1000 gallons of diesel fuel. Each container of stabilizer additive shall be labeled with the recommended effective concentration in both units.

3.3.2 Metal deactivator. The stabilizer additive shall contain sufficient metal deactivator so that when the stabilizer additive is mixed with diesel fuel at the recommended effective concentration (see 3.3.1), the concentration of metal deactivator active ingredient shall be not less than $2.5 \text{ g}/\text{m}^3$ and not more than $5.7 \text{ g}/\text{m}^3$. The metal deactivator shall be one of the following two types:

- a. N,N'-disalicylidene -1,2-propanediamine.
- b. N,N'-disalicylidene -1,2-cyclohexanediamine.

The use of metal deactivators other than the types and quantities specified shall require prior approval by the qualifying agency. The contractor shall report the type and amount of metal deactivator in the finished stabilizer additive.

3.3.3 Toxic products and formulations. The material shall have no adverse effect on the health of personnel when used for its intended purpose. Questions pertinent to this effect shall be referred by the procuring activity to the appropriate departmental medical service who will act as an advisor to the procuring activity. A Material Safety Data Sheet shall be provided in accordance with the requirements of FED-STD-313 and 29 CFR 1910.1200. When FED-STD-313 is at variance with the CFR, 29 CFR 1910.1200 shall take precedence, modify, and supplement FED-STD-313. (See 6.5).

3.4 Test fuels. The test fuel used to evaluate the stabilizer additive, except for biocidal activity and antirust properties, shall conform to the requirements of Caterpillar 1H2 Test Fuel, a diesel fuel widely used in evaluating the performance of crankcase lubricants. The specification for 1H2 Test Fuel can be found in STP 509A, part II, appendix F. The test fuels for biocidal activity and antirust properties shall be as specified in 4.6.14.2 and 4.6.15.1, respectively.

3.5 Reference formulation. The reference formulation shall consist of the test fuel described in 3.4, prefiltered through a 0.8 micron filter, to which both of the following products shall be added to give the concentrations indicated:

- a. du Pont Fuel Oil Additive FOA-15,^{1/} $71.3 \pm 0.5 \text{ g/m}^3$.
- b. Biobor JF,^{2/} $227 \pm 1 \text{ g/m}^3$.

3.6 Test formulation. The test formulation shall consist of the test fuel described in 3.4, prefiltered through a 0.8 micron filter, after which the contractor's stabilizer additive shall be added to give the recommended effective concentration (see 3.3.1).

3.7 Solubility. The stabilizer additive, at three times the recommended effective concentration (see 3.3.1), shall be readily and completely soluble in test fuel conforming to 3.4. There shall be no precipitation, cloudiness, or other insolubility when tested as specified in 4.6.2.

3.8 Compatibility. The stabilizer additive shall be compatible with all stabilizer additives currently qualified under this specification. There shall be no precipitation, cloudiness, or other evidence of incompatibility when tested as specified in 4.6.3.

3.9 Flash point. The flash point of the stabilizer additive shall not be less than 40 °C when tested in accordance with 4.6.4.

1/ FOA-15 is available from E.I. du Pont de Nemours and Co., Petroleum Laboratory, Wilmington, DE 19898.

2/ Biobor JF is available from U.S. Borax and Chemical Corporation, 3075 Wilshire Blvd., Los Angeles, California 90010.

3.10 Ash content. The ash content of the stabilizer additive shall not exceed 0.10 weight percent when determined in accordance with 4.6.5.

3.11 Minimum handling temperature. The minimum handling temperature of the stabilizer additive shall be reported in accordance with 4.6.6.

3.12 Diesel and gas turbine engine operation. The Government reserves the right to perform engine tests to determine the operational acceptability of stabilizer additives in diesel engines and in gas turbine engines authorized to operate on diesel fuel. Engine tests, if conducted, shall be in accordance with 4.6.7. Engine operation shall not be adversely affected and the post-test condition of the engine shall indicate no excessive deposits, wear, corrosion, or other deleterious effects which are attributed to the stabilizer additive.

3.13 Stabilizer additive storage stability. After storage for 12 months in accordance with 4.6.8, the stabilizer additive shall show no precipitation, layering, or other evidence of gross separation or degradation. Stabilizer additive representing the top half of the stored sample shall meet all requirements of this specification.

3.14 Filterability. The test formulation (see 3.6) shall be tested for filterability in accordance with 4.6.9. The filtration ratio shall not exceed 1.05.

3.15 Oxidation stability (accelerated). The test fuel (see 3.4), reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with 4.6.10. The results shall be reported in the certified test report (see 4.3.1).

3.16 High temperature stability. The test fuel (see 3.4), reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with 4.6.11. The results shall be reported in the certified test report (see 4.3.1).

3.17 Fuel storage stability. The test fuel (see 3.4), reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with 4.6.12. The results shall be reported in the certified test report (see 4.3.1).

3.18 Partition coefficient. The contractor shall report the value of the partition coefficient for the biocide component of the stabilizer additive as determined in accordance with 4.6.13.

3.19 Biocidal activity. The stabilizer additive shall be tested in accordance with 4.6.14. The test report shall contain the following data on each sample: test organisms, type of fuel, concentration of stabilizer additive, time since inoculation, and visual changes observed, including presence of granular particles, films on or between layers, mycelial mats, emulsions, flakes, color changes, turbidity, slime, and sludge. The lowest concentration of stabilizer additive which shows 100 percent kill or inhibition of all three test organisms shall be recorded as the effective biocidal concentration. This concentration,

when corrected for the partition coefficient (see 4.6.13) and the fuel:water ratio, shall be equal to or lower than the recommended effective concentration (see 3.3.1).

3.20 Antirust properties. The stabilizer additive shall be tested for antirust properties in accordance with 4.6.15. The rating of the test specimen shall be A to pass the test.

3.21 Workmanship. The finished stabilizer additive in bulk or container shall be uniform in appearance and visually free from grit, undissolved water, insoluble matter, or other adulteration.

3.22 Components. The stabilizer additive supplied in accordance with this specification shall consist of no more than two containers per complete fuel treatment package.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.1.1 Responsibility for compliance. All items must meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling in quality conformance does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to acceptance of defective material.

4.1.2 Stabilizer additive for addition to fuels. When a fuel contractor purchases the stabilizer additive for addition to fuels to be used by the Government, the manufacturer of the stabilizer additive shall certify to the purchaser that the product has been qualified under this specification. In addition, a test report showing compliance of the product with the requirements of 4.4 shall be supplied to the purchaser. Additional data may be required by the purchasing activity to establish compliance with this specification.

4.2 Classification of inspection. The inspection and testing of the stabilizer additive shall be classified as follows:

- a. Qualification inspection (see 4.3).
- b. Quality conformance inspection (see 4.4).
- c. Inspection of packaging (see 4.7).

4.3 Qualification inspection. Qualification inspection and testing shall consist of tests specified in 4.6.

4.3.1 Test report. A certified test report shall be forwarded to the activity responsible for qualification (see 6.4) before the qualification sample is supplied. The test report shall contain laboratory data showing the results required by 3.2, 3.3.1, 3.3.2, 3.7, 3.8, 3.9, 3.10, 3.11, 3.13, 3.14, 3.15, 3.16, 3.17, 3.18, 3.19, 3.20, and 3.21. In addition, stabilizer additive formulation data shall be supplied to the qualifying activity. This data shall include one of the following:

- a. Complete chemical name and percentage of each ingredient.
- b. Complete percentage composition of the stabilizer additive by chemical element, plus a general generic description of each ingredient showing type of functional groups.

The contractor shall furnish toxicological data and formulations required to evaluate the safety of the material for the proposed use, including a Material Safety Data Sheet prepared in accordance with 3.3.3.

4.3.1.1 Qualification sampling. Unless otherwise specified by the activity responsible for qualification, an initial 1-liter sample of finished stabilizer additive shall be submitted for evaluation in all of the tests except the storage stability and engine operation tests. If the product passes these tests, and additional sample of finished stabilizer additive will be requested for the storage stability, and engine tests. Samples shall be identified and packaged as required, and forwarded to the laboratory responsible for testing as designated in the letter of authorization from the activity responsible for qualification (see 6.4).

4.3.1.2 Regualification. Regualification will be required in the event of any change in composition or formulation, source of the stabilizer additive, its ingredients, or manufacturing sites; or if periodic verification determines noncompliance to requirements.

4.3.1.3 Retention of qualification. The retention of qualification of products approved for listing on the Qualified Products List shall be accomplished by a periodic verification to determine continued compliance of a supplier's product with the requirements of this specification. The verification intervals shall not exceed five years. Unless otherwise specified by the activity responsible for the qualified products list, verification of qualification may be made by certification.

4.4 Quality conformance inspection. Quality conformance inspection of a bulk lot of stabilizer additive shall consist of tests for conformance to requirements for solubility (see 3.7) flash point (see 3.9), ash (see 3.10), and property limits assigned to the product during qualification testing.

4.4.1 Inspection lot.

4.4.1.1 Bulk lot. A bulk lot is defined as an indefinite quantity of a homogeneous mixture of material offered for acceptance in a single isolated container, manufactured as a single isolated batch, or manufactured by a single plant run (not exceeding 5 days) through the same processing equipment with no change in ingredient material.

4.4.1.2 Packaged lot. A packaged lot is defined as an indefinite number of 55-gallon drums or smaller unit packages of identical size and type, offered for acceptance, and filled with a homogeneous mixture of material from a bulk lot.

4.4.2 Sampling. Each bulk or package lot of material shall be sampled for verification of product quality and compliance in accordance with ASTM D 4057.

4.5 Inspection. Inspection shall be in accordance with Method 9601 of FED-STD-791.

4.6 Test methods.

4.6.1 Identification tests. Identification tests shall be conducted in accordance with the following methods:

Density at 15 °C	ASTM D 1298
Viscosity at 0° and 40 °C	ASTM D 445
Neutralization number	ASTM D 664
pHr	Add 0.1 to 0.11 gram of stabilizer additive to 125 mL of D 974 titration solvent. Standardize apparatus and read pHr in accordance with Appendix X2 of D 974.
Metallic constituents	Emission or atomic absorption spectrograph.

4.6.2 Solubility test. Filter 1.0 liter of test fuel (see 3.4) through a 0.8 micron filter into a clean, round, flat-bottomed, transparent bottle or beaker. Add an amount of stabilizer additive sufficient to give not less than three times the recommended effective concentration (see 3.3.1). Stir or swirl the test fuel for a period not to exceed three minutes to dissolve the stabilizer additive. Check for insoluble materials by swirling the sample so a vortex is formed. All sediment and insoluble matter will accumulate on the bottom of the bottle directly beneath the vortex. The presence of visible sediment, water, haze, cloudiness, or other insolubility shall constitute failure of the test. If no insoluble matter is present, cover the sample and check again for insoluble matter by swirling the sample after it has stood at room temperature for 24 hours. The presence of visible sediment, water, haze, cloudiness, or other insolubility shall constitute failure of the test.

4.6.3 Compatibility. Prepare solutions of each stabilizer additive previously approved under this specification by dissolving each stabilizer additive in test fuel (see 3.4) at the recommended effective concentration (see 3.3.1). Mix each stabilizer additive solution with an equal volume of test formulation (see 3.6) in a clean, round, flat-bottomed, transparent bottle or beaker. Check for insoluble materials by swirling the sample until a vortex is formed. Sediment and insoluble matter will accumulate on the bottom of the flask directly beneath the vortex. If no insoluble matter is present, cover the sample and check it

again by swirling after it has stood at room temperature for 24 hours. The presence of visible haze, sediment, cloudiness, or other insolubility shall constitute failure of the test.

4.6.4 Flash point. The flash point of the stabilizer additive shall be determined in accordance with ASTM D 93. A flash point of less than 40 °C shall constitute failure of this test.

4.6.5 Ash content. The ash content of the stabilizer additive shall be determined in accordance with ASTM D 482. An ash content exceeding 0.10 weight percent shall constitute failure of this test.

4.6.6 Minimum handling temperature. The minimum handling temperature of the stabilizer additive shall be recorded as the temperature below which the stabilizer additive crystallizes or becomes too viscous for pouring, pumping, mixing, and blending operations as normally practiced in the petroleum industry.

4.6.7 Diesel and gas turbine engines operational test. The extent of engine testing shall be determined by the activity responsible for qualification based on the chemical composition of the stabilizer additive, evaluation of previous engine test data, if any, available from Government or commercial sources, and consideration of the number and type of military engines operating on diesel fuel. Fuel for engine testing shall conform to 3.4 or WV-F-800 and shall contain stabilizer additive at twice the recommended effective concentration. The engine test cycle shall be not less than 100 hours and shall conform to an established test method for which baseline data are available on the engine and lubricating oil used in the test. After completion of the engine test, the engine shall be disassembled and inspected. Evidence of excessive wear, deposits, corrosion, or other deleterious effects attributed to the stabilizer additive shall constitute failure of this test.

4.6.8 Stabilizer additive storage stability test. Two 1-liter amber glass bottles shall each be filled with 850 mL of the stabilizer additive and shall be tightly capped by means of a screw cap having a conical polyethylene liner. Each bottle shall be wrapped in a minimum amount of opaque packing material sufficient for protection against physical damage, but minimal in thermal insulation qualities. The wrapped bottles shall be inclosed in a tight wooden or metal box for further protection against breakage and sunlight. The crated samples shall be stored at ambient, outdoor conditions. The crated samples shall be kept off the ground and stored in an upright position under a canopy, open shed roof, or similar ventilated shelter for protection from direct sunlight and precipitation. The crated samples shall remain undisturbed for the specified period. One of the samples shall be stored for exactly 12 months and then removed for examination and testing; the other sample shall be stored for 12 months or less and may be removed for examination and testing any time at the option of the qualifying activity. Whenever a sample is removed for examination and testing, it shall be uncrated with minimum disturbance; the bottle shall not be shaken, inverted, or otherwise agitated. The contents of the bottle shall be inspected visually. Precipitation, separation into layers, or other evidence of gross separation shall constitute failure of this test. The presence or absence and the nature of such separation shall be recorded. The top half of the liquid sample shall be carefully removed by suction or siphoning into another bottle, without disturbing

the bottom half of the original sample. The top-half sample, after transfer to the second bottle, shall be shaken thoroughly and then used in laboratory testing, performed in accordance with 3.1.1. The bottom-half sample, in the original storage bottle, shall be retained for examination and possible additional testing to detect changes caused by storage.

4.6.9 Filterability test. The test formulation (see 3.6) shall be tested for filterability in accordance with Appendix, Test Method No. 1. Values greater than 1.05 shall constitute failure of this test.

4.6.10 Oxidation stability (accelerated). Duplicate samples of the test fuel (see 3.4), the reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with ASTM D 2274 for 16 hours, 32 hours, and 48 hours. Generation of more total insolubles in the test formulation than in the reference formulation, for two or more of the three test durations (16, 32 and 48 hours), shall constitute failure of the test. The mean value of the duplicate results shall be used in each of the pass/fail determinations.

4.6.11 High temperature stability test. Duplicate samples of the test fuel (see 3.4), the reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with Appendix, Test Method No. 2 for 1.5, 3.0, and 4.5 hour aging periods. Generation of more total insolubles in the test formulation than in the reference formulation, for two or more of the three test durations (1.5, 3.0, and 4.5 hours), shall constitute failure of the test. The mean value of the duplicate results shall be used in each of the pass/fail determinations.

4.6.12 Fuel storage stability test. Duplicate samples of the test fuel (see 3.4), the reference formulation (see 3.5), and the test formulation (see 3.6) shall each be tested in accordance with D 4625 for 8 and 16 week aging periods. Generation of more total insolubles in the test formulation than in the reference formulation, for one or both of the two test durations (8 and 16 weeks), shall constitute failure of the test. The mean value of the duplicate results shall be used in each of the pass/fail determinations.

4.6.13 Partition coefficient. Equal volumes of distilled water and test formulation (see 3.6) shall be shaken vigorously in a separatory funnel and allowed to equilibrate for not less than 24 hours at 25 ± 1 °C. By means of a suitable analytical method, the concentration of the biocide component of the stabilizer additive shall be determined in the fuel layer and the water layer. The partition coefficient shall be calculated by means of the equation:

$$P = \frac{[B]_f}{[B]_w}$$

Where P = partition coefficient

[B]_f = concentration of biocide in the fuel layer

[B]_w = concentration of biocide in the water layer

Report the value of the partition coefficient and the analytical method used to determine it.

4.6.14 Biocidal activity test. The stabilizer additive shall be tested for biocidal activity in accordance with Part I of SIM Special Publication No. 2. The test inocula shall include the following strains identified by American Type Culture Collection number: *Cladosporium resinae* 20495, *Pseudomonas aeruginosa* 33988, and *Candida tropicalis* 48138. Organisms are available from the American Type Culture Collection, 12301 Parklawn Drive, Rockville, MD 20852-1776, and may require an adaptation period of up to 30 days to demonstrate adequate growth in the hydrocarbon/nutrient test medium. The concentration range of stabilizer additive to be used in the tests shall be calculated in accordance with

4.6.14.1. Three concentrations of stabilizer additive shall be tested, using a hydrocarbon fuel as specified in 4.6.14.2. Each flask shall be examined at 30, 60, and 90 days after inoculation for evidence of growth or physical changes resulting from microbial growth. Evidence of growth in any of the test flasks containing the two highest of the three concentrations of stabilizer additive, at the examination times cited, shall constitute failure of the test. Results of the test at the lowest concentration of stabilizer additive shall be reported for information only.

4.6.14.1 Concentration of stabilizer additive for biocidal tests. The concentration of stabilizer additive to be used in biocidal activity tests shall be calculated taking into account the recommended effective concentration (REC) (see 3.3.1) and the partition coefficient (see 4.6.13). This calculation is required because the biocidal activity tests are conducted at a 1:1 ratio fuel to aqueous Bushnell-Haas solution, whereas the fuel:water ratio is typically 500:1 or greater in field applications. The concentration of stabilizer additive to be used in the test fuel for biocidal activity shall be the REC multiplied by the factor $(P + 1)/P$, where P is the partition coefficient determined in 4.6.13. For example, if the REC is 100 g/m³ and P = 0.5, the concentration of stabilizer additive to be used in the test fuel is $100 \times (0.5 + 1)/0.5$ or 300 g/m³. A minimum of two additional concentrations of stabilizer additive, one above and one below the calculated concentration, also shall be tested. The stabilizer additive shall be added only to the fuel phase, not to the aqueous phase. The complete stabilizer additive, not just the biocide component, shall be used in the biocidal activity tests.

4.6.14.2 Hydrocarbon test fuel for biocidal activity. The hydrocarbon test fuel used for biocidal activity testing shall be a kerosene-type mixture containing predominantly n-alkanes of chain length 8 to 18 carbons. Preliminary screening tests shall be conducted on the test fuel to demonstrate that it will support adequate growth of the three test organisms in the absence of biocide when incubated with an equal volume of Bushnell-Haas solution prepared in accordance with the appendix to SIM Special Publication No. 2. Adequate growth is not less than a 10-fold increase of the organism count in not more than 60 days of incubation at 25 °C.

4.6.15 Test for antirust properties. The test for antirust properties shall be conducted in accordance with NACE TM-01-72. The fuel used in this test shall be isooctane conforming to 4.6.15.1. A rating lower than A shall constitute failure of this test.

4.6.15.1 Test fuel for antirust properties. Isooctane conforming to TT-S-735, shall be freshly depolarized as follows: A glass chromatographic column or 1-liter separatory (Squibb) funnel is filled with silica gel to a height 20 cm above the stopcock, retaining the silica gel by means of a glass wool plug. (NOTE: Do not use stopcock grease). Approximately 3.8 liters of isooctane are passed through the silica gel bed by gravity, discarding the first 50 mL and collecting the remainder in a chemically clean glass container. The depolarized isooctane should be used within 1 week after treatment. The stabilizer additive to be tested shall be blended with the depolarized isooctane at the recommended effective concentration (see 3.3.1) to form the test fuel for 4.6.15.

4.7 Inspection of packaging.

4.7.1 Quality conformance inspection of pack.

4.7.1.1 Unit of product. For the purpose of inspection, a completed pack prepared for shipment shall be considered a unit of product.

4.7.1.2 Examination. Each container shall be examined to see how full the container is. Each container containing less material than specified in the contract shall be considered a defect. The samples shall be examined to determine conformance with the packaging, packing, palletization, and marking requirements of the contract. Any nonconformance with these requirements shall be considered a defect. Sampling shall be in accordance with MIL-STD-105, inspection level S-2. AQL shall be 1.0 percent defective for all defects.

5. PACKAGING

5.1 Packing and marking. The stabilizer additive contained in the size and type container specified (see 6.2) shall be packed and marked in accordance with MIL-STD-290. The degree of packing shall be as specified (see 6.2).

5.2 Packaging inspection. The inspection of these packaging requirements shall be in accordance with 4.7.

6. NOTES

6.1 Intended use. The stabilizer additive is intended to be added into diesel fuel to retard or prevent the formation of fuel deterioration products (i.e., gums, sludge, particulates) resulting from auto-oxidation processes, to reduce the potential for microbiological growth, and to provide for corrosion protection of fuel-wetted surfaces. Primarily, this product is for the treatment of fuel in (1) depot facilities where vehicles/equipment are in re-build or storage, (2) pre-positioned material at location involving storage of equipment partially or fully fueled, and (3) fuel stocks intended for intermediate (6-24 months) or long-term (25-60 months) storage.

6.2 Ordering data.

6.2.1 Acquisition requirements. Acquisition documents shall specify the following:

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- a. Title, number and date of the specification.
- b. Date of issue of DODISS applicable to this contract and exceptions thereto (see 2.1.1).
- c. Size and type of container required (see 5.1).
- d. Degree of packing required (see 5.1).
- e. Quantity of stabilizer additive required. The unit of purchase is one US gallon (3.785 liters) at 60 °F (15.6 °C).
- f. Type of stabilizer additive required (see 1.2).
- g. Component or components required (applies to type II only) (see 1.2.3).

6.3 Qualification. With respect to products requiring qualification, awards will be made only for products which are, at the time set for opening of bids, qualified for inclusion in Qualified Products List QPL-53021, whether or not such products have actually been so listed by that date. The attention of the contractors is called to these requirements, and manufacturers are urged to arrange to have the products that they propose to offer to the Federal Government tested for qualification in order that they may be eligible to be awarded contracts or purchase orders for the products covered by this specification. The activity responsible for the Qualified Products List is the USA Belvoir Research, Development, and Engineering Center, ATIN: STREE-VF, Ft. Belvoir, VA 22060-5606, and information pertaining to qualification of products may be obtained from that activity.

6.4 Subject term (key word) listing.

Additive, fuel
Antioxidant
Biocide
Corrosion inhibitor
Diesel fuel
Dispersant
Fuel oil, diesel
Metal deactivator
Military specifications
Stabilizer additive, diesel fuel

6.5 Material Safety Data Sheets. Contracting officers will identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with FED-STD-313. The pertinent government mailing addresses for submission of data are listed in Appendix B of FED-STD-313. One copy shall also be provided to the procuring activity.

6.6 Changes from previous issue. Asterisks or vertical lines are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

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APPENDIX

STABILIZER ADDITIVE, DIESEL FUEL

10. SCOPE

10.1 Scope. The test methods contained within this appendix are to determine whether the stabilizer additive procured under this specification conform to the requirements set forth.

APPENDIX

TEST METHOD NO. 1

DETERMINING THE FILTERABILITY OF DIESEL FUEL

100 SCOPE

100.1 This method is for determining the filterability characteristics of diesel fuel. It is intended to provide an indication as to whether the fuel will exhibit any potential filter plugging problem after addition of additives.

110 SUMMARY OF METHOD

110.1 In this method, 500 mL samples of clean and test fuel are filtered through a 0.8 micron filter disc. Fuel filterability is evaluated by the ratio of the filtering time for the test fuel to the filtering time for the clean fuel. As both fuels originate from the same source, clean refers to prefiltered whereas test refers to unfiltered prior to testing.

120 APPARATUS

120.1 The following equipment or its equivalent is used:

(SUGGESTED SUPPLIER)

- | | |
|--------------------------------------|------------------------|
| a. 01-092-25 Pressure/Vacuum Pump | Fisher Scientific. |
| b. 1000-mL Filter Flask | Fisher Scientific. |
| c. 500-mL Graduated Cylinder | Fisher Scientific. |
| d. Stop Watch | Fisher Scientific. |
| e. XX10-047-00 Pyrex Filter Holder | Millipore Corporation. |
| f. 0.8 Micron Filter Disc#AAWP-04700 | Millipore Corporation. |
| g. 1000-mL Beaker | Fisher Scientific. |

130 PRECAUTIONARY STATEMENT

130.1 It is very important to make all filtrations of any one sample at the same temperature. It is also important to keep the filter base clean. Clean it with petroleum solvents only. This should be done by pouring solvent into the outlet of the filter base and then forcing it thorough the base with air; then suck all the solvent form the base before using it to filter fuel.

140 PROCEDURE

140.1 Store all glassware and approximately 1020 mL of test fuel (see 3.4) at 25 ± 1 °C for 4 hours or until temperature equilibrium is attained.

140.2 Pour approximately 510 mL of the test fuel described in 3.4 into a 500-mL graduated cylinder.

APPENDIX

140.3 Install a 0.8 micron filter disc in the holder and assemble to the filter flask. Turn on the vacuum pump and allow to run for 10 or 15 seconds to build up full vacuum. Pour the 510 mL of untreated test fuel from the graduated cylinder into the filter bowl at a rate such that the filter bowl is kept full until the filtration is complete. Pour filtered fuel from the filtration flask into a clean 500-mL graduated cylinder until it reaches the 500 mL mark. Label as "untreated fuel" and set it aside. Discard the filter disc.

140.4 Install a clean 0.8 micron filter disc in the holder. Filter approximately 510 mL of test fuel as in the previous step. Pour the filtered fuel from the filtration flask into a 100-mL beaker and add a sufficient amount of the stabilizer additive being tested to bring the concentration of stabilizer additive to the recommended effective concentration (see 3.3.1). Swirl or stir to dissolve the stabilizer additive as completely as possible. After the additive has dissolved, pour the fuel from the 1000-mL beaker into a clean 500-mL graduated cylinder until it reaches the 500 mL mark. Label this container as "treated fuel". Note that this sample should be identical to the test formulation described in 3.6.

140.5 Install a clean 0.8 micron disc in the filter holder. Then pour 500 mL of untreated fuel from 130.3 into the filter exactly as before, but start the stop watch when the fuel hits the filter disc and stop it when all the fuel has been pulled through. (Starting and stopping the watch at the exact time is very important). The sample is to be maintained within ± 1 °C of the test temperature during this step.

140.6 Do not remove the filter disc, but pour out the fuel in the flask. Then filter and time 500 mL of treated fuel from 130.4 through the same filter disc that was used in step 130.5. The sample is to be maintained within ± 1 °C of the test temperature during this step.

150 CALCULATION

150.1 Divide time obtained in 130.6 by time obtained in 130.5, and round to the nearest 0.01. If the result is 1.00 the fuel was perfectly filterable. Values greater than 1.05 denote a potential problem which is due to additive-fuel incompatibilities. The higher the ratio value, the greater the problem in filterability.

APPENDIX

TEST METHOD NO. 2

HIGH TEMPERATURE (150 °C) STABILITY

OF DISTILLATE FUELS

200 SCOPE

200.1 This method determines relative stability of distillate fuels under high temperature aging conditions (150 °C) with limited air exposure.

210 SUMMARY OF METHOD

210.1 Measured volumes of distillate fuel are aged for 1.5, 3.0, and 4.5 hours at 150 °C in an open tube with air exposure. After aging and cooling, fuel is filtered and the amount of insoluble residue formed is estimated by determining the light reflectance of the filter pad.

210.2 As an alternative to the light reflectance method of determining residue in the aged fuel, the residue may be determined gravimetrically. An acceptable method is to use the Gooch crucible apparatus, filter paper, filtration procedure, and weighing procedure specified in ASTM D 2274 to replace 220.2, 220.3, and 260.6 through 260.10 of this test method. Determine the filterable insolubles only. Do not determine the adherent insolubles.

220 APPARATUS

220.1 Aging tubes, 2.5 x 20 cm, heavy wall tubes made of borosilicate glass.

220.2 Filter paper, glass fiber, 4.25 cm, 1.2 micron retention.

220.3 Membrane filter holder, to fit 47 mm membrane filters, vacuum source, and filtration flasks.

220.4 Heating bath, with liquid heating medium, thermostatically controlled and stirred to maintain the oil sample in the aging tube within 1.5 °C of 150 °C. It shall be large enough to hold aging tubes immersed in the heating liquid to a depth above the level of samples in the tubes. The bath and its location shall enable shielding of the samples from direct light during aging. The volume of oil in the bath and its heat recovery rate shall be such that the temperature of the heating medium does not drop more than 5 °C when the maximum number of aging tubes are inserted, and recovery to 150 °C does not require more than 15 minutes.

220.5 Paper reflectance meter^{1/}, black glass surface for calibration, and opaque white surface at least 10 x 20 cm in size.

^{1/} A Photovolt Paper Brightness Meter 670 complete with search unit W and black glass calibration standard, Catalog No. 00-572-32 is suitable and is available from Photovolt, 1115 Broadway, New York, NY 10010.

APPENDIX

230 REAGENTS

230.1 Hydrocarbon solvent, isooctane, ASTM knock test reference fuel grade. Adherent insolubles solvent, (equal parts of reagent grade toluene, methanol, and acetone).

240 PREPARATION OF SAMPLE

240.1 Samples for stability testing should be all-level samples obtained according to methods outlined in D 4057, Manual Sampling of Petroleum and Petroleum Products. Samples should be stored in metal cans, preferably with epoxy lining. Clear glass bottles are not acceptable as sample containers. Undue exposure to sunlight may produce erratic results. If a sample cannot be tested immediately, it should be stored under nitrogen at a temperature not higher than 10 °C. If samples are stored longer than one week, the date of sampling and date of testing shall be reported.

250 PREPARATION OF APPARATUS

250.1 Cleaning aging tubes - Clean new tubes by filling with cleaning solution^{2/} and allow to soak at least 2 hours. Rinse each scrupulously with tap water to remove all traces of acid, then with distilled water, followed by acetone, then dry. If compressed air is used it must be oil-free. Used tubes should be rinsed with adherent insolubles solvent, then with detergent, tap water, distilled water followed by acetone, then air dried.

250.2 Cleaning filter assembly - Membrane filter holders for which the filter rests on a sintered glass surface must be periodically cleaned with adherent insolubles solvent or cleaning solution followed by rinses as above.^{3/}

260 PROCEDURE

260.1 Adjust the heating bath to a temperature high enough to maintain the oil in the aging tubes at 150 ±1.5 °C.

260.2 Filter a 175-mL sample through a new filter paper into a clean vacuum flask using the membrane filter assembly.^{4/}

2/ A chromium-free cleaning solution, "Nochromix," is available from Godex Labs, 6 Varick Street, New York NY 10013.

3/ Partial blockage of the sintered glass surface can lead to uneven deposition of insoluble residues on the filter surface and reduce reliability of results. Contaminants may also be introduced during pre-filtration of fuel to give erratic results.

4/ When testing fuel with additives which may be adsorbed on the filter paper, the procedure may be modified so that the neat fuel is filtered first, then the additive is mixed with the filtered fuel.

APPENDIX

260.3 Measure three 50-mL portions of the filtered oil and decant into the aging tubes.

260.4 Place the uncapped sample tube in the heating bath for 1.5 hours ± 3 minutes, 3 hours ± 5 minutes, and 4.5 hours ± 7 minutes.

260.5 Remove each sample from the bath at the end of its aging time and allow to cool gradually to between 22 and 27 °C over a period of 2 to 4 hours in a location shielded from light.

260.6 Prepare a filtration assembly for each sample with a new filter paper and filter the fuel. Wash the aging tube with 3 small portions of isooctane and filter. Wash the filter assembly with isooctane and remove the funnel portion of the assembly. Wash the filter pad with several very small portions of isooctane and air dry.

260.7 Turn on the reflection meter and allow at least 30 minutes for warm-up. The "suppression" knob should be in the "off" position.

260.8 Place a new filter paper on the opaque white surface, place the search unit in the center of the filter and adjust the meter reading to 100 percent using the "sensitivity" knob.

260.9 Place the search unit in the center of the black glass standard and adjust the meter reading to 0 percent using the "zero" knob. Recheck the 100 percent adjustment against the new filter pad resting on the white surface and readjust if necessary.

260.10 Place the test filter pad on the white surface, center the unit on the filter, and record the meter value as "percent reflectance after test".^{5/}

270 REPORT

270.1 Report the sample identification and aging time at 150 °C.

270.2 Report the "percent reflectance after test". If the alternative method from 210.2 is used, multiply the weight of the filterable insolubles by 2.00 and report as "filterable insolubles, mg/100 mL".

270.3 If the test was not run within one week of fuel sampling, report sampling date and date of test.

5/ Dark fuels may stain the filter pads, resulting in lower reflectance ratings than due to insoluble residue alone.

APPENDIX

280 PRECISION

280.1 The repeatability and reproducibility of this test have not been determined. The following factors have been reported to affect results in some laboratories.

280.2 Sample storage time. Some fuels form degradation precursors during storage which markedly affect results, even when fuel appearance has not changed.

280.3 Filter paper porosity. The sensitivity of the method is changed when filters with different porosity or surface roughness are used.

280.4 Additive adsorption. Some additives are tenaciously adsorbed on the aging tubes and may affect results.

280.5 Heating bath design and location. Exposure to light during the aging step may affect results. High air flow rates across the open tubes during aging may affect the severity of the test.

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Army - ME
Navy - YD
Air Force - 68

Preparing activity:

Army - ME
Project 6850-0797

Review activities:

Army - AT, GL, SM
DLA - PS, GS

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Army - AV
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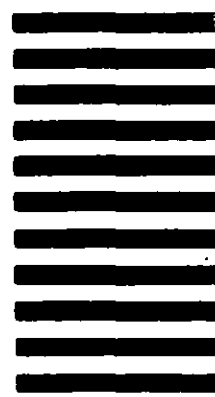
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1. DOCUMENT NUMBER MIL-S-53021A		2. DOCUMENT TITLE Stabilizer Additive, Diesel Fuel	
3a. NAME OF SUBMITTING ORGANIZATION		4. TYPE OF ORGANIZATION (Mark one)	
b. ADDRESS (Street, City, State, ZIP Code)		<input type="checkbox"/> VENDOR	
		<input type="checkbox"/> USER	
		<input type="checkbox"/> MANUFACTURER	
		<input type="checkbox"/> OTHER (Specify): _____	
5. PROPOSED CHANGES			
a. Recommended Wording:			
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7a. NAME OF SUBMITTER (Last, First, MI) - Optional		b. WORK TELEPHONE NUMBER (Include Area Code) - Optional	
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